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An Indentation Technique for Measuring Stresses in Tempered Glass Surfaces

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THE most practical way of strengthening glass plates is to introduce residual compressive stresses into the surfaces by physical and chemical tempering processes.[1–3] Traditionally, these stresses have been evaluated via the use of birefringence techniques,[4] which owe their widespread appeal to their inherent simplicity, their nondestructive nature, and their apparent capability of point-by-point determination of the entire stress profile across a given plate section. However, these optical techniques do have certain limitations. First, absolute values are not determined directly, for the birefringence technique measures only differences in principal stresses. Second, birefringence must be measured by passing a light beam through the edges and parallel to the surfaces of the plate without distortion. This approach places restrictions on the size and shape of the plate. Moreover, the typical glass plate contains optical inhomogeneities in the form of compositional striae parallel to the surfaces. Accordingly, stress measurements are often confined to a limited portion of the section, usually about the center plane of the plate, where stress gradients are minimal. Thus it is common practice to cite only the central (tensile) stress, averaged over the optical path length. But it is the compressive stress at the outer surfaces which is most pertinent to strength considerations: This quantity may be inferred from the measured value in conjunction with a presumed profile function (e.g. a parabolic function for thermally tempered plate, in which case the outer compression is extrapolated as twice the inner tension). Other conventional stress-sensing techniques which purport to measure surface stresses more directly, e.g. strain-gage techniques,[5] have met with little favor, mainly because of the degree of expertise required to make the measurements and because of difficulties in relating these measurements on treated plates to the stress-free state.

The present note describes a simple surface microprobe method which circumvents many of the shortcomings just mentioned. The procedure is based on the principle of indentation fracture,[6] in which the scale of microcracking around hardness impressions provides a measure of resistance to crack extension. In the present arrangement, the test surface is loaded with a standard diamond pyramid indenter. The "well-developed" deformation/fracture pattern is schematized in Fig. 1: "median" cracks of characteristic dimensions c (depth below surface) and c' (surface trace) expand as near half-pennies with increasing load P.[7,8,*] Comparative measurement of crack size on surfaces in the treated and untreated states might reasonably be expected to yield information on the residual (biaxial) compressive stress, σ_R.

A straightforward fracture-mechanics analysis establishes a quantitative basis for the technique. The stress intensity factor for the half-penny crack configuration of Fig. 1 can be written as the sum of 2 terms,

$$K=\chi P/c^{3/2}-2m\sigma_R(c/\pi)^{1/2} \qquad (1)$$

In the first term, representing the indentation driving force on the crack, χ is a dimensionless contact constant which incorporates details of the indenter/specimen contact[7]; in the second term, representing the residual resistance force, m is a dimensionless modification factor whose value is unity when free-surface effects and stress gradients over the prospective crack depth are neglected.[9] The crack remains in stable equilibrium during its growth if it satisfies the condition

$$K=K_c=\text{constant} \qquad (2)$$

where K_c is a material parameter.[10] Combination of Eqs. (1) and (2) then gives a relation with P and c as variables and K_c, m, and χ as

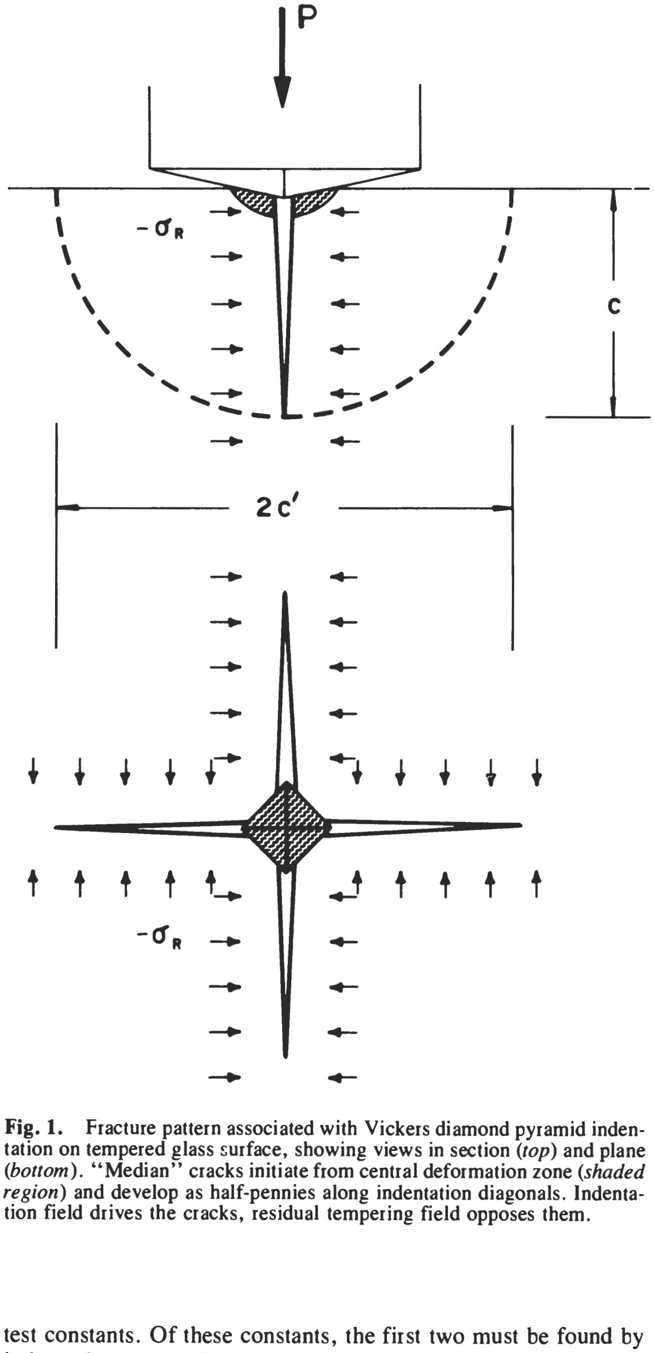

Fig. 1. Fracture pattern associated with Vickers diamond pyramid indentation on tempered glass surface, showing views in section (*top*) and plane (*bottom*). "Median" cracks initiate from central deformation zone (*shaded region*) and develop as half-pennies along indentation diagonals. Indentation field drives the cracks, residual tempering field opposes them.

test constants. Of these constants, the first two must be found by independent means, but χ may be determined directly from calibration tests on untempered plate. The general equilibrium equation may, accordingly, be written

$$(P/c^{3/2})_{\sigma_R}=(P/c^{3/2})_0(1+2m\sigma_R c^{1/2}/\pi^{1/2}K_c) \qquad (3)$$

where $(P/c^{3/2})_0=K_c/\chi=$ constant. A linear plot of $(P/c^{3/2})_{\sigma_R}$ vs $c^{1/2}$ would then enable σ_R for the tempered plate to be obtained from the slope.

Experimental results for soda-lime glass are illustrated in Fig. 2. The test specimens were slabs 30 by 30 by 6 mm cut from a single plate of annealed float glass. Data points in the figure represent indentation observations on 2 such slabs, one as-annealed and the other subjected to a thermal quenching treatment. The indenter force was delivered under dead-weight loading in dry N_2, and the corresponding equilibrium crack dimensions measured by travelling microscope either in profile at maximum impression or at the surface after indenter withdrawal. The patterns are considered to be well-developed when the characteristic dimension of cracking along the indentation diagonal is about twice that of the central deformation (Fig. 1); "low-load" data fall within the realm of "elastic/plastic" indentation fields[11] and, accordingly, tend to be

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*A second, "lateral" crack system, driven by mismatch stresses between the deformation zone and the surrounding elastic matrix, tends to cause some disruptive chipping at high loads (Ref. 6).

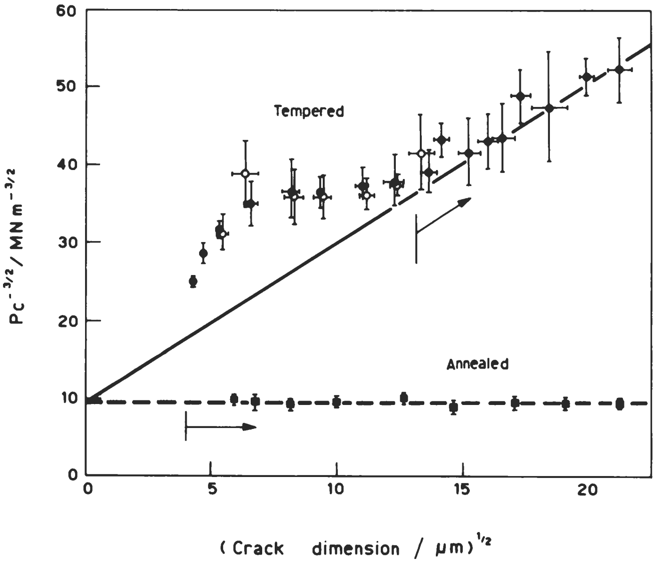

Fig. 2. Indentation fracture results for soda-lime glass plate in both tempered and annealed states. Each data point represents mean and standard deviation for ≥14 indentations at a specified load. *Open symbols* denote crack measurements in profile (c), *closed symbols* denote measurements from surface traces (c'). Experiments performed in dry N_2 atmosphere, loading time 15 s, such that kinetic effects in crack growth are minimal. Straight lines represent Eq. (3). *Arrows* indicate regions where crack patterns are "well-developed."

less reliable. Notwithstanding the uncertainties in the data, there is little to distinguish between the alternative modes of crack measurement. Straight lines in Fig. 2 are representations of Eq. (3): the (horizontal) broken line is a best fit to the indentation data at $\sigma_R=0$ (annealed specimen), from which we obtain $(P/c^{3/2})_0=(9.8\pm0.5)$ MN $m^{-3/2}$; the full line is an independent evaluation at $\sigma_R=(130\pm20)$ MPa (twice central tension measured optically through tempered specimen with compensator), with $K_c=(0.75\pm0.05)$ MN $m^{-3/2}$ (double-cantilever fracture measurements on soda-lime glass)[12] and $m=1$. The plotted results indicate that the indentation test can determine absolute surface stresses to considerably better than a factor of 2.

Since it needs only a conventional hardness testing facility, the present method compares favorably with its optical counterparts in economy of operation. Several advantages may be cited: Local variations in residual stress may be followed across a given surface; there is no restriction on material opacity, since remnant surface traces provide a measure of the crack dimension; and solids of irregular geometry may be examined. However, in plates where gradients of stress are severe (notably in chemically tempered glass), the modification factor m is expected to become a complex diminishing function of c,[13] thus invalidating the procedure just described. More important, the deliberate introduction of a crack into the surface of a brittle plate will inevitably lead to a degradation in strength, thereby precluding direct application of the method to potential load-bearing components; however, this limitation should not detract from applications to process control in which it is necessary only to test random controls in the mass production of ceramic components.

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